# PATENT COOPERATION TREATY

# **PCT**

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

POT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference				
Kear-2 PCT	Preliminar	ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)		
International application No.	International filing date (day/month/year)	Priority date (day/month/year)		
PCT/US00/22340	16 AUGUST 2000	16 AUGUST 1999		
International Patent Classification (IPC) Please See Supplemental Sheet.	or national classification and IPC			
Applicant RUTGERS, THE STATE UNIVERSI	тү			
2. This REPORT consists of a  This report is also accomp	panied by ANNEXES, i.e. sheets of the dec	red by this International Preliminary Article 36.  cription, claims and/or drawings which have agrectifications made before this Authority.		
(see Rule 70.16 and Section (see Rule 70.16 and Section (see Rule 70.16 and Section)	on our of the Administrative instructions in	nder the PCT).		
<del></del>	<del></del>			
3. This report contains indications	s relating to the following items:			
I X Basis of the repor	t			
II Priority				
III Non-establishmen	t of report with regard to povelty invest			
	t of report with regard to novelty, invent	ive step or industrial applicability		
	•			
V X Reasoned statement citations and explan	under Article 35(2) with regard to novelty, ations supporting such statement	inventive step or industrial applicability,		
VI Certain documents ci	ted			
VII Certain defects in the				
	on the international application	1		
III solutions	он ше шегнановаг аррисанов			
Date of submission of the demand	. Date of completion	of this report		
15 MARCH 2001 23 OCTOBER 2001				
Name and mailing address of the IPEA/U	S Authorized officer	0 2		
Commissioner of Patents and Trademark Box PCT Washington, D.C. 20231		BAREFORD My Call		
Facsimile No. (703) 305-3230	Telephone No. (70			
2004		-, 0001		

Form PCT/IPEA/409 (cover sheet) (July 1998)\*



International application No.

PCT/US00/22340

I. Ba	asis of	f the report	
1 With	h moand	d to the elements of the international application:*	
1		to the elements of the international application:*  nternational application as originally filed	
님		description:	
X	1	(For Americal)	
			, as originally filed
	pages	S S Sled with the Law C	, filed with the demand
	hap	s, filed with the letter of	
X		claims:	
_	pages	S (See Attached)	as originally filed
		s, as amended (together with any sta	atement) under Article 19
	pages	S	filed with the dament
	pages	s, filed with the letter of	, 11104 71141 415 45111111
X	the dra	rawings:	
		S (See Attached)	, as originally filed
	pages	S	filed with the demand
	pages	, filed with the letter of	
		equence listing part of the description:  (See Attached)	
			, as originally filed
	pages.	S	filed with the demand
	pages.	, filed with the letter of	
	the lang	tional application was filed, unless otherwise indicated under this item. nents were available or furnished to this Authority in the following language	der Rule 23.1(b)).
pren	iuminary	rd to any nucleotide and/or amino acid sequence disclosed in the international approximation was carried out on the basis of the sequence listing:	pplication, the international
		ned in the international application in printed form.	
		ogether with the international application in computer readable form.	
f	furnishe	ned subsequently to this Authority in written form.	
		ned subsequently to this Authority in computer readable form.	
ГТ	The state	atement that the subsequently furnished written sequence listing does not go beyo tional application as filed has been furnished.	ond the disclosure in the
Т		tement that the information recorded in computer readable form is identical to the un-	riten sequence listing has
	_	mendments have resulted in the cancellation of:	
Ļ	X th	he description, pagesNONE	
Ļ	Y	he claims, Nos 9 and 24	
L		he drawings, sheets/ <del>fig</del> NONE	ı
5. T			
·	hevond	port has been drawn as if (some of) the amendments had not been made, since they have the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**	ve been considered to go
* Replace in this and 70	cement sh s report ( 0.17).	sheets which have been furnished to the receiving Office in response to an invitation under a t as "originally filed" and are not annexed to this report since they do not contain at	imendments (Rules 70.16
**Any re	<u>eplacem</u>	ment sheet containing such amendments must be referred to under item 1 and annexed	ed to this report.



International application No.

PCT/US00/22340

V.	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;
	citations and explanations supporting such statement

#### 1. statement

Novelty (N)	Claims	1-8, 10-15 AND 28-33	YES
	Claims	16-23 AND 25-27	NO
Inventive Step (IS)	Claims	NONE	YES
	Claims	1-8, 10-23 AND 25-33	NO NO
Industrial Applicability (IA)	Claims	1-8, 10-23 AND 25-33	YES
	Claime	NONE	NO

#### 2. citations and explanations (Rule 70.7)

Claims 16-23 and 25-27 lack novelty under PCT Article 33(2) as being anticipated by Connolly et al (US 5120693).

Connolly teaches a method of making a powder, column 1, lines 5-10. As a ceramic-ceramic powder, it would be capable to be used in a thermal spray apparatus, column 2, lines 55-65. Micron-scale particles of a hard phase material are blended with nano-scale particles of a binder phase material to form a uniform powder mixture, see column 2, lines 55-65, column 10, lines 5-15 and column 5, lines 25-60. The binder phase material is a ceramic, silica, column 2, lines 60-65. The powder mixture is aggregated to bond the different particles together, column 5, lines 25-50.

claim 17: in the blending step, the particles are aggregated. column 5, lines 25-60.

claim 18: the particles are of different sizes. column 10, lines 5-20.

claims 19, 20: the particles are different in composition. column 10, lines 5-20 (note the difference between the micron particles and the binder particles).

claims 21, 22, 27: the particles are agglomerated by spray drying before aggregating by heat. column 5, lines 25-60.

claim 22: the final particle aggregate size can be 40 microns, for example. column 5, lines 45-50.

claim 23: The hard phase material is a ceramic. column 3, lines 20-40.

claims 25, 26: The micron scale particles can be 50 to 90 percent of the aggregate by weight, which would be inclusive of, for example, 70 volume percent. column 10, lines 5-20.

The Examiner notes that silica is considered to be a ceramic. For example, the Examiner notes claim 15, line 4 and claim 28, line 4, where SiO<sub>2</sub> (silica) is clearly indicated as a ceramic.

Claims 1-8, 10-15 and 28-33 an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of WO 97/18341 (hereinafter '341).

Connolly teaches all the features of these claims except (1) (Continued on Supplemental Sheet.)



International application No.

PCT/US00/22340

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

Claims 16 is objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because the claim are indefinite for the following reason(s):

claim 16, line 8, "from" should apparently be "form".



International application No.

PCT/US00/22340

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I · VIII

Sheet 10

#### CLASSIFICATION:

The International Patent Classification (IPC) and/or the National classification are as listed below: IPC(7): B32B 9/00; C22C 29/00; C23C 4/06 and US Cl.: 427/450, 451, 452, 453, 455, 456; 75/230, 236, 240, 244, 245, 246; 428/552, 565, 688, 689, 539.5; 419/10, 12, 13, 14, 18; 106/286.8, 287.34

### I. BASIS OF REPORT:

This report has been drawn on the basis of the description, page(s) 1-17, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:

This report has been drawn on the basis of the claims, page(s) NONE, as originally filed. page(s) NONE, as amended under Article 19. page(s) NONE, filed with the demand. and additional amendments:

Pages 18-23, filed with the letter of 07 September 2001.

This report has been drawn on the basis of the drawings, page(s) 1-6, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:
NONE

This report has been drawn on the basis of the sequence listing part of the description: page(s) NONE, as originally filed.
pages(s) NONE, filed with the demand.
and additional amendments:
NONE

### V. 2. REASONED STATEMENTS - CITATIONS AND EXPLANATIONS (Continued):

the thermal spraying of the feedstock to form a coating (claims 1, 29), (2) the selective melting during spraying (claim 13), (3) the cooling rate (claim 14), (4) the particular materials of the particles (claims 15, 28, 30, 31, 33).

'341 teaches the thermal spraying of agglomerated particles to form wear resistant coatings. page 5, lines 10-20. The particles are agglomerated from nanostructured particles. page 5, lines 10-20. '341 further teaches that when spraying nanoparticle agglomerates the nanoparticle grains and the binder will all rapidly dissolve, while when spraying larger particles surrounded by a matrix phase, the matrix will melt, but the larger particle will undergo little size change and result in a porous coating, see page 9, lines 10-30 and figures 4A and 4B. '341 teaches that the materials sprayed can be WC/Co agglomerates, for example, see page 8, lines 10-25.

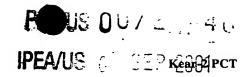
It would have been obvious to one of ordinary skill in the art at the time the invention was made to modify Connolly to use the particles for thermal spraying as suggested by '341 with an expectation of producing desired ceramic coatings, because Connolly teaches providing agglomerated ceramic powders, and '341 teaches that it is conventional to thermal spray agglomerated powders. It further would have been obvious to use the micron/nano scale particles as taught by Connolly when selective melting is desired during spraying, such as to provide a porous coating, given the selective melting taught to be conventional by '341 when using particles larger than nanoparticle sized. As to the cooling rate, it would have been obvious to control the cooling rate to provide the desired structure, given the teaching of Connolly to control the temperature to prevent destroying the crystal structure of the particles during processing (see column 5, lines 25-40). Further, it would have been obvious that the particles could be made of other ceramic/metal or ceramic/ceramic combinations based on the coating requirements given the teaching in '341 of the variety of materials that can be agglomerated together to form thermal spraying particles.

The Examiner notes that silica is considered to be a ceramic. For example, the Examiner notes claim 15, line 4 and claim 28, line 4, where SiO<sub>2</sub> (silica) is clearly indicated as a ceramic. The Examiner notes that Connolly would be considered analogous art, because it is reasonably pertinent to the problem of agglomerating particles. The reference to '341 has been



International application No.

INTERNATIONAL PRELIMINARY EXAMINATION 1037 03.17	PCT/US00/22340
Supplemental Box (To be used when the space in any of the preceding boxes is not sufficient)	
Continuation of: Boxes I - VIII	Sheet 11
provided as to the conventionality of using agglomerated particles is thermal spray co	pating processes.
Claims 1-8, 10-15 and 28-33 meet the criteria set out in PCT Article 33(2) as to not does not teach the thermal spraying of the particular coating.	velty, because the prior art to Connolly
Claims 1-8, 10-23 and 25-33 meet the criteria set out in PCT Article 33(4), as to in provide a desirable method, particles and coating for providing desirable abrasion re-	dustrial applicability, because the claims esistant coatings.
NEW CITATIONS	
·	



## **CLAIMS**

What is claimed is:

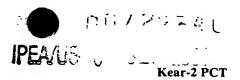
1. A method for producing an abrasion resistant coating composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture;

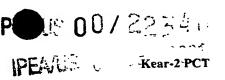
aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming a feedstock powder comprised of aggregated particles; and

thermal spraying the feedstock powder of particle aggregates onto a substrate thereby forming the abrasion resistant coating thereon, the coating composed of the micron-scale particles of the hard phase material fused together with the binder phase material.

- 2. The method according to claim 1, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.
  - 3. The method according to claim 2, wherein the particles are different sizes.
- 4. The method according to claim 3, wherein the particles are different in composition.



- 5. The method according to claim 2, wherein the particles are different in composition.
- 6. The method according to claim 1, further comprising the step of agglomerating the powder mixture formed in the blending step prior to performing the aggregating step.
- 7. The method according to claim 6, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 8. The method according to claim 1, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 10. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 11. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.
- 12. The method according to claim 1, wherein the aggregating step is performed by heat treating.
- 13. The method according to claim 1, wherein during the thermal spraying step the nano-scale particles of the binder phase material are selectively melted, the melted particles



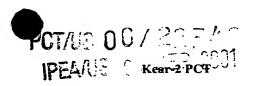
filling pore spaces between heated and softened ones of the micron-scale particles, thereby effectively binding the micron-scale particles together and densifying the coating.

- 14. The method according to claim 13, wherein during the thermal spraying step the melted particles experience a cooling rate which generates one of an amorphous nanocrystalline or microcrystalline binder phase.
- 15. The method according to claim 1, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
- 16. A method of making a feedstock powder for use in producing thermal spray abrasion resistant coatings composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture; and

aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming particle aggregates which from the feedstock powder.

17. The method according to claim 16, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.



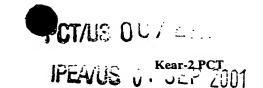
- 18. The method according to claim 17, wherein the particles are different sizes.
- 19. The method according to claim 18, wherein the particles are different in composition.
- 20. The method according to claim 17, wherein the particles are different in composition.
- 21. The method according to claim 16, further comprising the step of agglomerating the powder mixture formed in the blending step prior performing the aggregating step.
- 22. The method according to claim 21, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 23. The method according to claim 16, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 25. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 26. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.

- 27. The method according to claim 16, wherein the aggregating step is performed by heat treating.
- 28. The method according to claim 16, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
  - 29. An abrasion resistant coating comprising:

nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles; and

micron-scale particles of a hard phase material fused together with the binder phase material.

- 30. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/metal material system.
- 31. The coating according to claim 30, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal.



- 32. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/ceramic material system.
- 33. The coating according to claim 32, wherein the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.



From the INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To: PAUL A. SCHWARZ DUANE, MORRIS & HECKSCH 100 COLLEGE ROAD WEST, S PRINCETON, NJ 08540	ER LLP UITE 100 ECEIVE NOV 27 2001	INTERN	PCT TION OF TRANSMITTAL OF NATIONAL PRELIMINARY AMINATION REPORT (PCT Rule 71.1)
Applicant's or agent's file reference		IMI	PORTANT NOTIFICATION
Kear-2 PCT	International filing d	ata (day/month/year)	Priority Date (day/month/year)
International application No.	International Hing d	ate (uity/ monthly Jeur)	
PCT/US00/22340	16 AUGUST 200	00	16 AUGUST 1999
Applicant RUTGERS, THE STATE UNIVE	RSITY		

- The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith 1. the international preliminary examination report and its annexes, if any, established on the international
- A copy of the report and its annexes, if any, is being transmitted to the International Bureau for 2. communication to all the elected Offices.
- Where required by any of the elected Offices, the International Bureau will prepare an English translation of 3. the report (but not of any annexes) and will transmit such translation to those Offices.

#### REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume  $\Pi$  of the PCT Applicant's Guide.

Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Authorized officer

KATHERINE A. BAREFORD / WILL

Washington, D.C. 20231

Telephone No. (703) 308-0661

Facsimile No. (703) 305-3230 Form PCT/IPEA/416 (July 1992)\*

2/16/02 30 mo. not placedue

No920-00014





International application No.

### PCT/US00/22340

	isis of the report				
1. With	regard to the elements of the international application:*				
	the international application as originally filed				
$\mathbf{x}$	the description:				
	pages (See Attached)				
	pages	_ , filed with the demand			
	pages, filed with the letter of				
x	the claims:				
		, as originally filed			
	pages, as amended (together with any s				
	pages	_ , filed with the demand			
	pages, filed with the letter of				
x	the drawings:				
	pages (See Attached)	, as originally filed			
	pages	_ , filed with the demand			
	pages, filed with the letter of				
	the sequence listing part of the description:				
X	pages (See Attached)	as originally filed			
	pages	filed with the demand			
	pages, filed with the letter of	,			
41	th regard to the language, all the elements marked above were available or furnished to this A international application was filed, unless otherwise indicated under this item.				
Th	ese elements were available or furnished to this Authority in the following language	which is:			
	the language of a translation furnished for the purposes of international search (	under Rule 23.1(b)).			
	the language of publication of the international application (under Rule 48.3(b)).				
	the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and				
"	or 55.3).				
3. W	ith regard to any nucleotide and/or amino acid sequence disclosed in the international	l application, the international			
pr	preliminary examination was carried out on the basis of the sequence listing:				
▎└	contained in the international application in printed form.				
	filed together with the international application in computer readable form.				
furnished subsequently to this Authority in written form.					
	furnished subsequently to this Authority in computer readable form.				
	The statement that the subsequently furnished written sequence listing does not go be interesting to filed her been furnished.	beyond the disclosure in the			
	international application as filed has been furnished.  The statement that the information recorded in computer readable form is identical to the	writen sequence listing has			
_	been furnished.	When sequence issues into			
4. X	The amendments have resulted in the cancellation of:				
	X the description, pages NONE				
	To a second seco				
	the claims, 140s.				
	The drawings, sheetsing	, ,			
5.	This report has been drawn as if (some of) the amendments had not been made, since the	y have been considered to go			
* D	beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).** placement sheets which have been furnished to the receiving Office in response to an invitation use	nder Article 14 are referred to			
in.	nacement sneets which have been jurnished to the receiving Office in response to an invitation at this report as "originally filed" and are not annexed to this report since they do not conto t 70.17).	in amendments (Rules 70.16			
	y replacement sheet containing such amendments must be referred to under item 1 and ar	nexed to this report.			





International application No.

PCT/US00/22340

V.	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability;
	citations and explanations supporting such statement

#### 1. statement

Novelty (N)	Claims Claims	1-8, 10-15 AND 28-33 16-23 AND 25-27	YES NO
Inventive Step (IS)	Claims Claims	NONE 1-8, 10-23 AND 25-83	YES NO
Industrial Applicability (IA)	Claims Claims	1-8, 10-23 AND 25-33 NONE	YES

## 2. citations and explanations (Rule 70.7)

Claims 16-23 and 25-27 lack novelty under PCT Article 33(2) as being anticipated by Connolly et al (US 5120693).

Connolly teaches a method of making a powder. column 1, lines 5-10. As a ceramic-ceramic powder, it would be capable to be used in a thermal spray apparatus. column 2, lines 55-65. Micron-scale particles of a hard phase material are blended with nano-scale particles of a binder phase material to form a uniform powder mixture. see column 2, lines 55-65, column 10, lines 5-15 and column 5, lines 25-60. The binder phase material is a ceramic, silica column 2, lines 60-65. The powder mixture is aggregated to bond the different particles together. column 5, lines 25-50.

claim 17: in the blending step, the particles are aggregated. column 5, lines 25-60.

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claim 23: The hard phase material is a ceramic. column 3, lines 20-40.

claims 25, 26: The micron scale particles can be 50 to 90 percent of the aggregate by weight, which would be inclusive of, for example, 70 volume percent. column 10, lines 5-20.

The Examiner notes that silica is considered to be a ceramic. For example, the Examiner notes claim 15, line 4 and claim 28, line 4, where SiO, (silica) is clearly indicated as a ceramic.

Claims 1-8, 10-15 and 28-33 an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of WO 97/18341 (hereinafter '341).

Connolly teaches all the features of these claims except (1) (Continued on Supplemental Sheet.)





International application No. PCT/US00/22540

# VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

Claims 16 is objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because the claim are indefinite for the following reason(s):

claim 16, line 8, "from" should apparently be "form".





International application No.

PCT/US00/22340

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I · VIII

Sheet 10

### CLASSIFICATION:

The International Patent Classification (IPC) and/or the National classification are as listed below: IPC(7): B32B 9/00; C22C 29/00; C23C 4/06 and US C1.: 427/450, 451, 452, 453, 455, 456; 75/230, 236, 240, 244, 245, 246; 428/552, 565, 688, 689, 539.5; 419/10, 12, 13, 14, 18; 106/286.8, 287.34

#### I. BASIS OF REPORT:

This report has been drawn on the basis of the description, page(s) 1-17, as originally filed. page(s) NONE, filed with the demand. and additional amendments: NONE

This report has been drawn on the basis of the claims, page(s) NONE, as originally filed. page(s) NONE, as amended under Article 19. page(s) NONE, filed with the demand. and additional amendments: Pages 18-23, filed with the letter of 07 September 2001.

This report has been drawn on the basis of the drawings, page(s) 1-6, as originally filed. page(s) NONE, filed with the demand. and additional amendments: NONE

This report has been drawn on the basis of the sequence listing part of the description: page(s) NONE, as originally filed. pages(s) NONE, filed with the demand. and additional amendments: NONE

V. 2. REASONED STATEMENTS - CITATIONS AND EXPLANATIONS (Continued):

the thermal spraying of the feedstock to form a coating (claims 1, 29), (2) the selective melting during spraying (claim 13), (3) the cooling rate (claim 14), (4) the particular materials of the particles (claims 15, 28, 30, 31, 33).

'341 teaches the thermal spraying of agglomerated particles to form wear resistant coatings. page 5, lines 10-20. The particles are agglomerated from nanostructured particles. page 5, lines 10-20. '341 further teaches that when spraying nanoparticle agglomerates the nanoparticle grains and the binder will all rapidly dissolve, while when spraying larger particles surrounded by a matrix phase, the matrix will melt, but the larger particle will undergo little size change and result in a porous coating. see page 9, lines 10-30 and figures 4A and 4B. '341 teaches that the materials sprayed can be WC/Co agglomerates, for example. see page 8, lines 10-25.

It would have been obvious to one of ordinary skill in the art at the time the invention was made to modify Connolly to use the particles for thermal spraying as suggested by '341 with an expectation of producing desired ceramic coatings, because Connolly teaches providing agglomerated ceramic powders, and '341 teaches that it is conventional to thermal spray agglomerated powders. It further would have been obvious to use the micron/nano scale particles as taught by Connolly when selective melting is desired during spraying, such as to provide a porous coating, given the selective melting taught to be conventional by '341 when using particles larger than nanoparticle sized. As to the cooling rate, it would have been obvious to control the cooling rate to provide the desired structure, given the teaching of Connolly to control the temperature to prevent destroying the crystal structure of the particles during processing (see column 5, lines 25-40). Further, it would have been obvious that the particles could be made of other ceramic/metal or ceramic/ceramic combinations based on the coating requirements given the teaching in '341 of the variety of materials that can be agglomerated together to form thermal spraying particles.

The Examiner notes that silica is considered to be a ceramic. For example, the Examiner notes claim 15, line 4 and claim 28, line 4, where SiO<sub>2</sub> (silica) is clearly indicated as a ceramic. The Examiner notes that Connolly would be considered analogous art, because it is reasonably pertinent to the problem of agglomerating particles. The reference to '341 has been

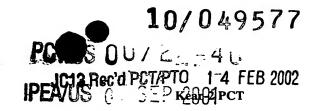




International application No.
PCT/US00/22340

Supplemental Box (To be used when the space in any of the preceding boxes is not sufficient)	
Continuation of: Boxes I - VIII	Sheet 11
provided as to the conventionality of using agglomerated particles is thermal spray co	pating processes.
Claims 1-8, 10-15 and 28-33 meet the criteria set out in PCT Article 33(2) as to not does not teach the thermal spraying of the particular coating.	velty, because the prior art to Connolly
Claims 1-8, 10-23 and 25-33 meet the criteria set out in PCT Article 33(4), as to in provide a desirable method, particles and coating for providing desirable abrasion r	dustrial applicability, because the claims esistant coatings.
NEW CITATIONS	
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### **CLAIMS**

What is claimed is:

1. A method for producing an abrasion resistant coating composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture;

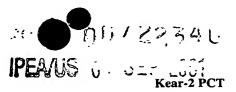
aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming a feedstock powder comprised of aggregated particles; and

thermal spraying the feedstock powder of particle aggregates onto a substrate thereby forming the abrasion resistant coating thereon, the coating composed of the micron-scale particles of the hard phase material fused together with the binder phase material.

- 2. The method according to claim 1, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.
  - 3. The method according to claim 2, wherein the particles are different sizes.
- 4. The method according to claim 3, wherein the particles are different in composition.







- 5. The method according to claim 2, wherein the particles are different in composition.
- 6. The method according to claim 1, further comprising the step of agglomerating the powder mixture formed in the blending step prior to performing the aggregating step.
- 7. The method according to claim 6, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 8. The method according to claim 1, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 10. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 11. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.
- 12. The method according to claim 1, wherein the aggregating step is performed by heat treating.
- 13. The method according to claim 1, wherein during the thermal spraying step the nano-scale particles of the binder phase material are selectively melted, the melted particles





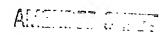
filling pore spaces between heated and softened ones of the micron-scale particles, thereby effectively binding the micron-scale particles together and densifying the coating.

- 14. The method according to claim 13, wherein during the thermal spraying step the melted particles experience a cooling rate which generates one of an amorphous nanocrystalline or microcrystalline binder phase.
- 15. The method according to claim 1, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
- 16. A method of making a feedstock powder for use in producing thermal spray abrasion resistant coatings composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

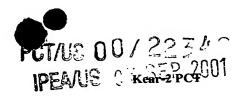
blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture; and

aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming particle aggregates which from the feedstock powder.

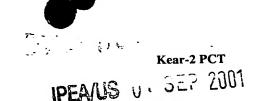
17. The method according to claim 16, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.







- 18. The method according to claim 17, wherein the particles are different sizes.
- 19. The method according to claim 18, wherein the particles are different in composition.
- 20. The method according to claim 17, wherein the particles are different in composition.
- 21. The method according to claim 16, further comprising the step of agglomerating the powder mixture formed in the blending step prior performing the aggregating step.
- 22. The method according to claim 21, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 23. The method according to claim 16, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 25. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 26. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.



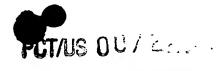
- 27. The method according to claim 16, wherein the aggregating step is performed by heat treating.
- 28. The method according to claim 16, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
  - 29. An abrasion resistant coating comprising:

nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles; and

micron-scale particles of a hard phase material fused together with the binder phase material.

- 30. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/metal material system.
- 31. The coating according to claim 30, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal.





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- 32. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/ceramic material system.
- 33. The coating according to claim 32, wherein the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.





CLAIMS PERIACED BY
AND 34 AND T

What is claimed is:

1. A method for producing an abrasion resistant coating composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture;

aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming a feedstock powder comprised of aggregated particles; and

thermal spraying the feedstock powder of particle aggregates onto a substrate thereby forming the abrasion resistant coating thereon, the coating composed of the micron-scale particles of the hard phase material fused together with the binder phase material.

- 2. The method according to claim 1, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.
  - 3. The method according to claim 2, wherein the particles are different sizes.
- 4. The method according to claim 3, wherein the particles are different in composition.



- 5. The method according to claim 2, wherein the particles are different in composition.
- 6. The method according to claim 1, further comprising the step of agglomerating the powder mixture formed in the blending step prior to performing the aggregating step.
- 7. The method according to claim 6, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 8. The method according to claim 1, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 10. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 11. The method according to claim 1, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.
- 12. The method according to claim 1, wherein the aggregating step is performed by heat treating.
- 13. The method according to claim 1, wherein during the thermal spraying step the nano-scale particles of the binder phase material are selectively melted, the melted particles



filling pore spaces between heated and softened ones of the micron-scale particles, thereby effectively binding the micron-scale particles together and densifying the coating.

- 14. The method according to claim 13, wherein during the thermal spraying step the melted particles experience a cooling rate which generates one of an amorphous nanocrystalline or microcrystalline binder phase.
- 15. The method according to claim 1, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
- 16. A method of making a feedstock powder for use in producing thermal spray abrasion resistant coatings composed of a ceramic/metal material system or a ceramic/ceramic material system, the method comprising the steps of:

blending micron-scale particles of a hard phase material with nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles to form a uniform powder mixture; and

aggregating of the powder mixture to bond the nano-scale particles to the micron-scale particles thereby forming particle aggregates which from the feedstock powder.

17. The method according to claim 16, wherein in the blending step the micron-scale particles of the hard phase material are arranged in particle aggregates.



- 18. The method according to claim 17, wherein the particles are different sizes.
- 19. The method according to claim 18, wherein the particles are different in composition.
- 20. The method according to claim 17, wherein the particles are different in composition.
- 21. The method according to claim 16, further comprising the step of agglomerating the powder mixture formed in the blending step prior performing the aggregating step.
- 22. The method according to claim 21, wherein the agglomerating step is performed by spray drying and the particle aggregates are each about 5 to 50 microns in diameter.
- 23. The method according to claim 16, wherein the hard phase material includes one of a ceramic or a ceramic/metal composite.
- 25. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises between 50 and 90 volume percent of the blended powder mixture.
- 26. The method according to claim 16, wherein the micron-scale particles of the hard phase material comprises 70 volume percent of the blended powder mixture.



- 27. The method according to claim 16, wherein the aggregating step is performed by heat treating.
- 28. The method according to claim 16, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal and the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.
  - 29. An abrasion resistant coating comprising:

nano-scale particles of a binder phase material selected from the group consisting of metal particles, ceramic particles, and ceramic/metal composite particles; and

micron-scale particles of a hard phase material fused together with the binder phase material.

- 30. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/metal material system.
- 31. The coating according to claim 30, wherein the ceramic/metal material system is selected from the group consisting of WC/Co, Cr<sub>3</sub>C<sub>2</sub>/NiCr, TiC/Fe, metal boride/metal, and metal nitride/metal.



- 32. The coating according to claim 29, wherein the binder and hard phase materials are composed of a ceramic/ceramic material system.
- 33. The coating according to claim 32, wherein the ceramic/ceramic material system is selected from the group consisting of Al<sub>2</sub>O<sub>3</sub>, YSZ, Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>, ZrO<sub>2</sub>/MgO, and Cr<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub>.

# ATENT COOPERATION TRUITY

### From the INTERNATIONAL BUREAU

### **PCT**

### **NOTIFICATION OF ELECTION**

(PCT Rule 61.2)

To:

Commissioner **US Department of Commerce United States Patent and Trademark** Office, PCT 2011 South Clark Place Room CP2/5C24 Arlington, VA 22202 **ETATS-UNIS D'AMERIQUE** 

Date of mailing (day/month/year) 22 June 2001 (22.06.01)

in its capacity as elected Office

International application No. PCT/US00/22340

Applicant's or agent's file reference Kear-2 PCT

International filing date (day/month/year) 16 August 2000 (16.08.00)

Priority date (day/month/year) 16 August 1999 (16.08.99)

**Applicant** 

KEAR, Bernard, H. et al

1. The designated Office is hereby notified of its election made:									
	X in the demand filed with the International Preliminary Examining Authority on:								
	15 March 2001 (15.03.01)								
	in a notice effecting later election filed with the International Bureau on:								
2.	The election X was								
	was not								
	made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).								

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland

**Authorized officer** 

Zakaria EL KHODARY

Facsimile No.: (41-22) 740.14.35

Telephone No.: (41-22) 338.83.38



From the INTERNATIONAL SEARCHING AUTHORITY

PCT To: PAUL A. SCHWARZ **BUCHANAN INGERSOLL, P.C.** 650 COLLEGE ROAD EAST, 4TH FLOOR PRINCETON, NJ 08540 NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT OR THE DECLARATION (PCT Rule 44.1) Date of Mailing (day/month/year) 08.1AN 2001 Applicant's or agent's file reference FOR FURTHER ACTION See paragraphs 1 and 4 below Kear-2 PCT International filing date (day/month/year) International application No. PCT/US00/22340 16 AUGUST 2000 Applicant RUTGERS. THE STATE UNIVERSITY The applicant is hereby notified that the international search report has been established and is transmitted herewith. Filing of amendments and statement under Article 19: The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46): The time limit for filing such amendments is normally 2 months from the date of transmittal of the international search report; however, for more details, see the notes on the accompanying sheet. Where? Directly to the International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35 For more detailed instructions, see the notes on the accompanying sheet. The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith. With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that: the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices. no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made. The applicant is reminded of the following: 4. Further action(s): Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in rules 90 bis 1 and 90 bis 3, respectively, before the completion of the technical preparations for international publication. Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later) .

Name and mailing address of the ISA/US	Authorized officer
Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	KATHERINE A. BAREFORD WILL WAR
	C: Telephone No. (703) 308-0661
Pacsimile No. (703) 305-3230	100 page 100. (700) 200 0001

priority date or could not be elected because they are not bound by Chapter II.

Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the



# **PCT**

## INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference  Kear-2 PCT	FOR FURTHER see Notification of Transmittal of International Search Report ACTION (Form PCT/ISA/220) as well as, where applicable, item 5 below		Transmittal of International Search Report  1) as well as, where applicable, item 5 below.					
International application No.	International filing date	(day/month/year)	(Earliest) Priority Date (day/month/year)					
PCT/US00/22340	16 AUGUST 2000		16 AUGUST 1999					
Applicant RUTGERS, THE STATE UNIVERSITY								
This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.								
·	This international search report consists of a total of sheets.							
X It is also accpanied by a copy of each prior art document cited in this report.								
<ol> <li>Basis of the report         <ul> <li>a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.</li> <li>the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).</li> </ul> </li> </ol>								
b. With regard to any nucleotide was carried out on the basis o	and/or amino acid sequen f the sequence listing:	ce disclosed in the in	nternational application, the international search					
contained in the internation		orm.						
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furnished subsequently to t	his Authority in written fo	rm.						
furnished subsequently to t	his Authority in computer	readable form.						
the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.  the statement that the information recorded in computer readable form is identical to the written sequence listing has								
furnished. 2. Certain claims were foun	James and able (See Box	T\						
2. Certain claims were foun 3. Unity of invention is lack		. 1).						
4. With regard to the title,	ung (occ box 11).							
X the text is approved as sub	mitted by the applicant.							
the text has been established	ed by this Authority to rea	d as follows:						
5. With regard to the abstract,								
the text is approved as sub	mitted by the applicant.							
the text has been establishe Box III. The applicant may search report, submit com	y, within one month from to ments to this Authority.	he date of mailing of	this international					
6. The figure of the drawings to be	6. The figure of the drawings to be published with the abstract is Figure No. 1							
as suggested by the applic	ant.		None of the figures.					
X because the applicant faile	d to suggest a figure.							
because this figure better	characterizes the invention		•					

Box III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)

The technical features mentioned in the abstract do not include a reference sign between parentheses (PCT Rule 8.1(d)).

### **NEW ABSTRACT**

A thermal spray method for the fabrication of ceramic/metal and ceramic/ceramic hardcoatings for wear applications. The method makes use of feedstock powder, composed of micron-scale aggregates of hard phase material particles that are either mixed or coated with a readily fusible nano-scale binder phase material (12). Thus, during thermal spraying (14), the nanostructured material undergoes rapid melting while the aggregated material is heated but not necessarily melted. A dense coating is formed when the molten nano-material fills the available pore spaces between the heated and softened aggregates, providing a strong and tough matrix for the consolidated material. Optimal wear properties are achieved when the volume fraction of aggregated particles is high, typically in the range of 0.5 - 0.9. Aggregated material may be composed of one, two or more particles of different sizes and/or compositions, with particle size distribution that gives high packing density for the hard phase.

rniernational application No. PCT/US00/22340

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Electronic o	data base consulted during the international search (	name of data base and, where practicable	, search terms used)		
C. DOC	UMENTS CONSIDERED TO BE RELEVANT				
Category*	Category* Citation of document, with indication, where appropriate, of the relevant passages				
X  Y	US 5,120,693 A (CONNOLLY et al) line 55 through column 3, line 20 and	16-20  1-33			
X  Y	US 5,880,382 A (FANG et al) 09 Marc 60 and column 8. lines 40-60.	16-20  1-33			
Y	WO 97/18341 A1 (THE UNIVERSI May 1997. See pages 7-10.	FY OF CONNECTICUT) 22	1-33		
Purthe	er documents are listed in the continuation of Box C	See patent family annex.			
A* document to be the control of the automatical document the process of the process o	ment published prior to the international filing date but later than briority date claimed ctual completion of the international search ER 2000	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention  X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone  Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art  &* document member of the same patent family  Date of mailing of the international search report			
Commissione Box PCT Washington, acsimile No.		Authorized officer  KATHERINE A. BAREFORD  Telephone No. (703) 308-0661	Rupi Wolle		

# A. CLASSIFICATION OF SUBJECT MATTER: US CL $\,:\,$

427/450, 451, 452, 453, 455, 456; 75/230, 236, 240, 244, 245, 246; 428/552, 565, 688, 689, 539.5; 419/10, 12, 13, 14, 18; 106/286.8, 287.34

B. FIELDS SEARCHED Minimum documentation searched Classification System: U.S.

427/450, 451, 452, 453, 455, 456; 75/230, 236, 240, 244, 245, 246; 428/552, 565, 688, 689, 539.5; 419/10, 12, 13, 14, 18; 106/286.8, 287.34